भारतीय मानक
Indian Standard

IS 1448 [P : 118] : 2019

पेट्रोलियम एवं इसके उत्पादों के परीक्षण पद्धतियां

[पी : 118]

अल्ट्रावॉयलेट स्पेक्ट्रोमेट्री द्वारा एविएशन टरबाईन ईंधन में नेफथेलीन हायड्रोकार्बन की परीक्षण

(पहला पुनरीक्षण)

Methods of Test for Petroleum and its Products

[P : 118]

Naphthalene Hydrocarbons in Aviation Turbine Fuel by Ultraviolet Spectrophotometry

(First Revision)

ICS 75.080

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Methods of Sampling and Test for Petroleum, its Products, Gaseous Fuels and Lubricants Sectional Committee, PCD 01

FOREWORD

This Indian Standard [P : 118] (First Revision) was adopted by the Bureau of Indian Standards on recommendation of the Methods of Sampling and Test for Petroleum, its Products, Gaseous Fuels and Lubricants Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1985 as an adoption of ASTM D 1840-81 to determine the naphthalene content in jet fuels containing not more than 5 percent of such components and having end points below 315°C.

Improvements have taken place over time and the applicability of the methodologies are evolved with direct dilution (procedure B) with minimized error relative to three stage dilution process. The range of concentrations used in the inter laboratory tests which established the precision statements for this test method were 0.03 to 4.25 volume percent for procedure A, and 0.08 to 5.6 volume percent for procedure B. The direct dilution method however emits the possibility of carryover of deviations from three stage successive dilution procedure.

This first revision of the standard has been brought out to incorporate modifications found necessary in the light of the use of the test method. The significant modifications of this revision include:

a) Parallel method (Method B) for direct dilution of the sample and determination of naphthalene thereof is introduced; and

b) The spectroscopic grade isooctane as solvent is favored in comparison to cyclohexane.

Considerable assistance is taken from ASTM D 1840-07 and API Research Project 44 – Description and Analysis, (Author - Richard E. Voeltz), while preparing this revision.

This standard may involve hazardous materials, operation and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use for specific warning statements.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 118]

NAPHTHALENE HYDROCARBONS IN AVIATION TURBINE FUEL BY ULTRAVIOLET SPECTROPHOTOMETRY

(First Revision)

1 SCOPE

This standard [P : 118] test method covers the determination of the total concentration of naphthalene, acenaphthene, and alkylated derivatives of these hydrocarbons in jet fuels containing not more than 5 percent of such components and having end points below 315°C, by ultraviolet spectrophotometry.

NOTE — This test method should not be used to measure and describe the properties of the materials, products or assemblies of molecules other than Aviation Turbine Fuel (ATF) samples, although the scope can be extended on account of precision and repeatability.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provision of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
IS 1448 [P : 105] : 1981	Methods of test for petroleum and its products: [P : 105] Ultraviolet absorbance and absorptivity of petroleum products

3 OUTLINE OF THE METHOD

The total concentration of naphthalene in the jet fuels is determined by measurement of the absorbance at 285 nm of a solution of the fuel at known concentration.

4 SIGNIFICANCE

This test method for the naphthalene hydrocarbons is one of the groups of tests used to assess the combustion characteristics of aviation turbine fuels of the kerosene boiling range. The naphthalene hydrocarbon content is determined because when naphthalene burned, tend to have a relatively larger contribution to a sooty flame, smoke and thermal radiation than mono aromatics.

5 DEFINITIONS

5.1 Transmittance — The molecular property of the sample that determines the transportability of electromagnetic radiation expressed by:

$$T = P/Po$$

where,

P = radiant power passing through the sample, and

Po = radiant power incident upon the sample.

5.2 Absorbance, A — The molecular property of a substance to absorb the electromagnetic radiation and is expressed by absorbance:

$$A = \log_{10} (1/T) = -\log_{10} T$$

where,

T = Transmittance

5.2.1 *Absorptivity, a* — Specific property of substance to absorb the electromagnetic radiation per unit sample concentration and path length, expressed by:

$$a = A/bc$$

where,

A = Absorbance;

b = sample cell path length; and

c = concentration.

5.2.2 Quantitative ultraviolet analyses are based upon the absorption law, known as Beer's law. The law states that the absorbance of a homogenous sample containing an absorbing substance is directly proportional to the concentration of the absorbing substance at a single wavelength expressed by:

$$A = a\,b\,c$$

Where,

A = absorbance,

a = absorptivity,

b = sample path length, and

c = concentration, the quantity of sample contained in a unit quantity of solvent.

5.3 Concentration, c — The quantity of sample contained in a unit quantity of solvent, that is, the quantity of aromatic hydrocarbon in grams per litre of isooctane.

5.4 Sample Cell Path Length, b — It is the distance in centimeter, measured in the direction of electromagnetic radiation wave between the surface of the specimen on which the electromagnetic radiation is incident and the surface of the specimen from which it is emergent.

5.4.1 The distance does not include the thickness of the cell in which the specimen is contained.

6 INTERFERENCES

6.1 Saturated hydrocarbons, olefins, thiophenes, and alkyl or cycloalkyl derivatives of benzene will not interfere.

6.2 Interferences can occur from phenanthrenes, dibenzothiophenes, biphenyls, benzothiophene, and anthracene, if present. The end point limitation of 315°C will minimize this interference except for benzothiophene and biphenyls. The contribution to measured naphthalene content by the presence of 1 percent of such interfering compounds can be estimated from Table 1.

Table 1 Error in Percentage of Naphthalenes Caused by 1 Percent Interfering Compound

(*Clause* 6.2)

Sl No.	Types of Interfering Compound	Error in Percentage of Naphthalenes Caused by 1 Percent Interfering Compound
(1)	(2)	(3)
i)	Phenanthrenes	2
ii)	Dibenzothiophenes	2
iii)	Biphenyls	1
iv)	Benzothiophenes	0.6
v)	Anthracene	0.1

7 APPARATUS

7.1 UV Spectrophotometer

The UV spectrophotometer shall be capable to measure the absorbance of the solution in the spectral range of 240-300 nm, with a spectral slit width of 1 nm or less. Wavelength measurement shall be repeatable and known to be accurate within 0.1 nm or less as measured by mercury emission line at 253.63 nm or the absorption of either holmium oxide glass at 287.5 nm or holmium oxide solution at 287.1nm. Absorbance at the 0.4 absorbance level in the spectral region between 240-300 nm, the absorbance measurement shall be repeatable within ± 0.5 percent or better. In the absorbance range encompassing 0.2 to 0.8, the photometric accuracy shall not differ by more than ± 0.5 percent, of sample whose absorbance has been established by a standardizing laboratory.

NOTE — The secondary standards provided by the manufacturers should be traceable with respect to NIST primary standards for checking the wavelength accuracy and photometric accuracy. These materials can be used to verify the spectrophotometer performance provided they have been recalibrated periodically as recommended by manufacturer.

7.2 Vitreous Silica Cells

Two having a path length of 1.00 ± 0.005 cm or better. The distance does not include the thickness of the cell in which the sample is contained.

7.3 Pipette, Class A

7.4 Balance

The balance capable of weighing 100 g to nearest 0.0001 g. The balance shall be accurate to ± 0.0002 g at a 100 g load.

8 SOLVENT

8.1 Spectroscopic Solvent

Spectroscopic grade 2,2,4 trimethyl pentane (Iso-octane) or cyclohexane. The isooctane solvent that has a transmission greater than 90 percent compared to distilled water over the spectral range 240 to 300 nm should be used for dilution.

NOTES

1 Spectroscopic grade isooctane is available commercially*. Technical grade iso-octane is a satisfactory basestock for preparation of spectroscopic solvent. Allow about 4 or 5 l of technical grade iso-octane to percolate through a column of 50.8-76.2 mm in diameter and 0.6-0.9 mm in depth containing activated silica gel 200 mesh size. Collect only the portion of the solvent that has transmission compared to distilled water greater than 90 percent over the entire spectral range from 240 to 300 nm. Store in a scrupulously clean glass stoppered bottles and always keep covered. In general, it will be best to use a fresh portion of silica gel in preparing a new batch of solvent. However, the gel can be reactivated by pouring 500ml of acetone through the column, draining, drying by suction and heating the gel in thin layers in an oven at 400°C until the white color is restored. Activated silica gel is stored in closed containers.

2 Cyclohexane may be used as an alternative to isooctane in this procedure and is commercially available in spectroscopic grade.

3 Warning — Isooctane is extremely flammable and harmful if inhaled.

8.2 Solvent for Cleaning the Cell

Acetone or ethyl alcohol are used to clean the volumetric flask.

8.3 Selection of Path Length

The use of sample cell path length of 1cm is recommended.

9 CALIBRATION AND STANDARDIZATION

Instead of calibration of the spectrophotometer with known naphthene, the average absorptivity of the C_{10} to C_{13} naphthalene at 285 nm may be taken as 33.7 litres per gram centimeter. The data used to calculate this average are given in Table 2.

Table 2 Data to calculate Average Absorptivity

(*Clause* 9)

Sl No. (1)	Compound (2)	API Sl No. (3)	Absorptivity L/g.cm (4)
i)	Naphthalene	605	28.5
ii)	1-methyl Naphthalene	539	32.0
iii)	2-methyl Naphthalene	572	22.9
iv)	1,2-dimethyl Naphthalene	215	37.3
v)	1,3-dimethyl Naphthalene	216	36.4
vi)	1,4-dimethyl Naphthalene	217	43.5
vii)	1,5- dimethyl Naphthalene	218	54.0
viii)	1,6 dimethyl Naphthalene	219	36.4
ix)	1,7 dimethyl Naphthalene	220	36.0
x)	1,8 dimethyl Naphthalene	221	46.0
xi)	2,3- dimethyl Naphthalene	222	22.0
xii)	2,6- dimethyl Naphthalene	226	21.3
xiii)	2,7- dimethyl Naphthalene	224	23.5
xiv)	1-iso-propyl Naphthalene	203	31.7
xv)	Average absorptivity		33.7

NOTES

1 This data is issued by API Research Project – 44 and API Serial numbers are mentioned for guidance only.

2 The arithmetic average of the above absorptivities is 33.7. The reliability of the average absorptivity as a measure of selected individual naphthalenes may be estimated from Table 2.

10 PROCEDURE

10.1 Procedure A — Serial Dilution

Prepare three dilutions of the sample as follows.

10.1.1 *First Dilution*

If the sample is more volatile than isooctane, then take a 10-15 ml of spectroscopic isooctane in a clean, dry glass stoppered 25 ml volumetric flask and added 1 g of the sample to the flask mix well and dilute the volume with solvent. If the sample is less volatile in isooctane, then weigh out approximately 1g of sample in the 25 ml volumetric flask, dilute to volume with spectroscopic solvent, and mix thoroughly and make the volume up to the mark with spectroscopic grade solvent.

10.1.2 *Second Dilution*

Pipette 5.00 ml of the first dilution into 50 ml glass stoppered volumetric flask, dilute the volume with spectroscopic grade solvent up to the mark and mix thoroughly.

10.1.3 *Third Dilution*

Pipette 5 ml of the second dilution to 50 ml volumetric flask in the same manner as in **10.1.2**.

10.1.4 *Measurement of the Absorbance*

Transfer portions of the third dilute solution into the sample cell of the spectrophotometer. Check the windows of the absorption cell and make certain that they are cleaned. Measure the absorbance. Read the absorbance of the sample as compared to spectroscopic grade isooctane at 285 nm.

NOTES:

1 The user may use alternative procedure B if preferred.

2 Check carefully actions on handling and cleaning of cells and glassware, instrument adjustments and method of absorbance measurement.

3 The dilution of the sample should be performed in a regulated manner that the absorbance reading falls within a range of 0.2 to 0.8 for maximum reproducibility of the results. To accomplish this, it may be necessary to use an alternative third dilution than the one specified in **10.1.3** such as 10 ml of the second dilution to 25 ml with solvent.

10.2 Procedure B - Alternative 100 ml Dilution

The incorporation of the single dilution procedure has been included as an alternative procedure to reduce test time, glassware, cleaning and dilution errors.

10.2.1 *Sample Preparation*

Add an appropriate amount of sample to a clean, dry tared 100 ml volumetric flask, record the weight to the nearest 0.0001 g. Dilute to the mark with the spectroscopic grade isooctane, stopper and mix thoroughly.

10.2.2 *Determination of the Cell Correction*

Measure and record the absorbance of the spectroscopic grade isooctane filled sample cell as compared to the spectroscopic isooctane filled the solvent cell.

10.2.3 Refer to Table 3 for lists of sample weights associated with naphthalene concentration that gives absorbance reading between 0.2 to 0.8.

Table 3 Estimated Sample Weight and Volume to take for the Volume Percent Naphthalene Content of the Sample in Single Dilution Procedure to keep the Absorption Values between 0.2 to 0.8 Units (Assuming a Density of 0.8)

(*Clause* 10.2.3)

Sample Volume(ml)	Sample Weight (mg)	Volume Percentage Naphthalene for Expected Absorbance of 0.2 units	Volume Percentage of Naphthalene for Expected Absorbance of 0.8
0.050	40	1.2	4.8
0.075	60	0.8	3.2
0.100	80	0.6	2.4
0.150	120	0.4	1.6
0.200	160	0.3	1.2
0.300	240	0.2	0.8

NOTE — A micropipette is a convenient tool for adding an appropriate volume. If the fuel density is not known at the time of sample preparation, use 0.8 as an approximation.

11 CALCULATIONS

11.1 Calculate the mass percentage of the naphthalenes in the sample as follows:

$$\text{Naphthalene, mass percent} = \frac{(A \times K)}{(33.7 \times W)} \times 100$$

where,

A = corrected absorbance (observed absorbance – cell correction) of the dilution measured

For procedure A, in **10.1** using serial dilution:

K = equivalent volume of the solvent in litres, if the dilution had been made in a single step.

For the first, second and third dilutions, the value of K shall be 0.025, 0.25 and 2.5 respectively.

For suggested alternative third dilution k = 0.625

For procedure B in 10.2 using 100 ml dilution:

K = 0.1

W = grams of the sample used, and

33.7 = the average absorptivity of C_{10} to C_{13} naphthalenes in litres per gram centimeter

11.2 Calculate the volume percentage of naphthalenes as follows:

$$\text{Naphthalene, volume percent} = M \times \frac{B}{C}$$

Where,

M = percentage of naphthalene by mass;

B = relative density of the total fuel (15°C/15°C); and

C = relative density of the naphthalene (15°C/15°C) = 1.0.

12 REPORT

Numerical values of volume percentage of naphthalene are reported to nearest 0.01 percent.

NOTE — REFERENCE SPECTRA — Absorptivities of individual naphthalene hydrocarbons at 285 nm are derived from data in the API catalog of ultraviolet spectral data issued by research project 44 as given in Table 2.

13 PRECISION AND BIAS

13.1 Precision

The precision of procedure A was determined by from the test results for samples covering 0.03 to 4.25 volume percent naphthalene. The precision for procedure B was determined from 0.08 to 5.6 volume percent naphthalene.

13.1.1 For procedure A result of duplicate tests shall not differ by more than following amounts:

Repeatability	$(x + 1.094) \times 0.0133$
Reproducibility	$(x + 1.094) \times 0.0623$

Where,

x is the average of two results.

13.1.2 For procedure B, result of duplicate tests shall not differ by more than following amounts:

Repeatability	$0.0041\, x^{0.657}$
Reproducibility	$0.0936\, x^{0.657}$

Where,

x is the average of two results

13.2 Bias

Bias cannot be determined for the procedure in this test method for measuring naphthalene hydrocarbons because the absorptivity will vary with the composition of the individual naphthalene derivatives in the samples.

NOTE — Although cyclohexane can be used as a solvent instead of iso-octane, recent studies have shown that naphthalene values are found higher than those produced by isooctane. A survey of users has shown isooctane to be general use, and the use of cyclohexane should be dropped.

ANNEX A

PRECAUTIONARY STATEMENTS

A-1 ISOOCTANE

a) Danger — Extremely flammable;
b) Harmful if inhaled, vapor may cause flash fire;
c) Keep away from heat, sparks, and open flame;
d) Keep container closed;
e) Use with adequate ventilation;
f) Avoid buildup of vapours and eliminate all sources of ignition, especially non explosion proof electrical apparatus, and heaters;
g) Avoid prolonged breathing of vapors and spray mist; and
h) Avoid prolonged and repeated skin contact.

A-2 CYCLOHEXANE

a) Danger — Extremely flammable;
b) Harmful if inhaled, vapor may cause flash fire;
c) Keep away from heat, sparks, and open flame;
d) Keep container closed;
e) Use with adequate ventilation;
f) Avoid buildup of vapors and eliminate all sources of ignition, especially non explosion proof electrical apparatus, and heaters;
g) Avoid prolonged breathing of vapors and spray mist; and
h) Avoid prolonged and repeated skin contact.

A-3 ACETONE

a) Danger — Extremely flammable;
b) Harmful if inhaled, vapor may cause flash fire;
c) Keep away from heat, sparks, and open flame;
d) Keep container closed;
e) Use with adequate ventilation;
f) Avoid buildup of vapors and eliminate all sources of ignition, especially non explosion proof electrical apparatus, and heaters;
g) Avoid prolonged breathing of vapors and spray mist; and
h) Avoid contact with eyes and skin.

A-4 ETHYL ALCOHOL

a) Danger — Cannot be made nontoxic;
b) Denatured — Cannot be made nontoxic;
c) Harmful if inhaled, vapor may cause flash fire;
d) Keep away from heat, sparks, and open flame;
e) Keep container closed;
f) Use with adequate ventilation;
g) Avoid buildup of vapors and eliminate all sources of ignition, especially non explosion proof electrical apparatus, and heaters;
h) Avoid prolonged breathing of vapors and spray mist; and
j) Avoid prolonged and repeated skin contact.

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